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A METHOD FOR OBTAINING CARBON MONOXIDE
[Verfahren zum Gewinnen von Kohlenmonoxid]

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The invention concerns a method for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane, in which

- a) the gas stream is cooled and partially condensed,
- b) the first hydrogen-rich fraction that accumulates in the partial condensation is removed,
- c) the carbon monoxide-rich condensate, which contains methane and nitrogen and is obtained in the partial condensation, is sent to a hydrogen stripping column,
- d) in said column, separation into a second hydrogen-rich fraction and another carbon monoxide-rich fraction takes place, and
- e) said carbon monoxide-rich fraction, containing methane and nitrogen, is separated at low temperature into a highly pure carbon monoxide fraction and a fraction essentially containing methane and nitrogen.

Because of increased requirements on the purity of the fractionation products, the ever-increasing importance of plant operating costs and the constant improvement of the available thermodynamic data, a continuing technical change has occurred in recent years in H_2/CO fractionation.

As before, the steam reformer may be mentioned as the principal supplier of a gas stream that essentially contains hydrogen, carbon monoxide and methane and may possibly be contaminated by nitrogen. However, the gasification of heavy oil with oxygen, thus a partial oxidation, has come to be important as an H_2/CO supplier in recent years, assuming a cheap oxygen source.

Most of the carbon monoxide produced in this way is used in the production of formic and acetic acid. Another buyer is found in the polycarbonate chemistry, which requires high purity phosgene as a raw material, where the phosgene in turn requires carbon monoxide of extremely high purity. Here the methane content of the carbon monoxide must be less than 10 mol-ppm, and the water content must be less

* [Numbers in right margin indicate pagination of the original text.]

than 1000 mol-ppm. The hydrogen produced in obtaining pure CO is used for various hydrogenation purposes, optionally after subsequent fine purification.

An overview of the methods used to produce carbon monoxide and hydrogen as a byproduct can be found in the article by R. Fabian in LINDE-Berichte aus Technik und Wissenschaft ["LINDE Reports"], No. 55, 1984, pp. 38-42, and by Dr. R. Berninger in LINDE-Berichte aus Technik und Wissenschaft, No. 62, 1988, pp. 18-23.

Frequently, the specialist is faced with the problem of high nitrogen contents in the incoming gas stream. Higher nitrogen contents arise in particular when natural gas or nitrogen-containing refinery gases are used. The nitrogen contained in the incoming stream is particularly problematic in low-temperature fractionation.

Various possibilities for solving the problems that are caused by nitrogen are known:

First, one can use an oil or coal gasification process in which as a rule lower nitrogen contents are present, instead of the fractionation of a nitrogen-containing gaseous input in certain cases. Apart from the fact that with this conduct of the process one must switch to a different feed stock, it has the disadvantage that it is not necessarily the cheapest process. Thus, gasification processes as a rule are too costly in the case of plants having a carbon monoxide production of less than about 5000 Nm³/H [STP].

Another possibility is simply to accept the content of nitrogen in the carbon monoxide as long as it remains within justifiable limits. However, this basically simple conduct of the process has the crucial disadvantage that the conduct of the step processing the carbon monoxide becomes difficult and costly. Another possibility is to subject the gaseous feed to a nitrogen separation before the fractionation. However, this solution requires a second low temperature fractionation before fractionation with the corresponding necessary prepurification steps. For this reason the method involves considerable additional expenditure.

Finally, it would still be possible to obtain the carbon monoxide from the gas stream by washing processes instead of by low temperature fractionation. One such method is known, for example, from Figure 6 and the relevant description in the last-mentioned LINDE Reports. In this process the carbon monoxide contained in the cooled incoming gas is washed with very cold methane in a methane washing column. The condensate of this methane washing column is then sent to a hydrogen stripping column, in which the dissolved hydrogen is heated. The mixture, which is now free of hydrogen and essentially consists of carbon monoxide and nitrogen, is separated into a nitrogen/carbon monoxide fraction and a heavy methane fraction in a connected rectification column. The latter fraction is at least partially pumped back up to the pressure of the unfractionated gas, and it is used as a washing agent in the methane washing column. The resulting nitrogen/carbon monoxide fraction is then separated into a nitrogen fraction and a carbon monoxide product fraction in an additional rectification column.

However, the integration of a nitrogen/carbon monoxide separation within a hydrogen/carbon monoxide separation process involves considerable additional costs. Also, the required energy expenditure rises considerably, while the carbon monoxide yield decreases by a few percent. Compared to an otherwise necessary nitrogen separation from natural gas before a steam reformer, this solution nevertheless is in many cases the more profitable and simpler, if the nitrogen content in the carbon monoxide product must be limited.

If the feed gas does not contain any nitrogen or only small amounts of nitrogen, the conduct of the process as a rule becomes considerably simpler; one example of this is shown in Figure 7 of the article that has already been mentioned a number of times, in which a condensation process with preliminary pressure exchange absorption is described. In this case, hydrogen is removed from the carbon monoxide-rich condensate that is formed in a partial condensation, in a hydrogen stripping column, and methane is removed in a connected rectification column.

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The task of this invention is to specify a method for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains water, carbon monoxide and methane that is, for one thing, technically simpler and, for another, is cheaper than the known methods.

This is achieved in accordance with the invention by the fact that

f) the carbon monoxide-rich fraction that contains methane and nitrogen coming from the hydrogen stripping column is sent to a first rectification column,

g) in said rectification column, a separation into a nitrogen-rich and a carbon monoxide-rich fraction containing methane takes place,

h) the nitrogen-rich fraction is withdrawn,

i) the carbon monoxide-rich fraction containing methane is sent to a second rectification column, and

k) in said rectification column, a separation into the highly pure carbon monoxide product fraction and into a fraction essentially containing methane takes place.

The method in accordance with the invention for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane manages without the previously used methane wash. Instead, the carbon monoxide-rich fraction that essentially contains methane and nitrogen and is essentially free of hydrogen is sent to two successively arranged rectification columns. In this case, separation into a nitrogen-rich fraction and a carbon monoxide-rich fraction that contains methane takes place in the first of the two rectification columns. Then the remaining methane is removed in the second of the two rectification columns and, in this way, a highly pure carbon monoxide product fraction that has the desired product purity is obtained.

While the method described at the start and in Figure 6 of the said LINDE Reports has a total of four (rectification) columns, only three (rectification) columns are required in the method in accordance with

the invention. A reduction of investment costs for a plant of this kind results from this fact. In addition, the method in accordance with the invention is a technically much simpler process.

In a further development of the method in accordance with the invention it is proposed that at least one partial stream of the carbon monoxide product fraction, after condensation and cold-producing expansion, be used for the refrigeration required for the process. Alternatively, the refrigeration required for the process can take place by means of a closed (mixture) refrigeration cycle. In this case, nitrogen in particular is suitable as the refrigerating agent for the closed refrigeration cycle.

The method in accordance with the invention, as well as further developments thereof, is explained in more detail by means of Figures 1-3 and Tables A through C [sic].

Here:

Figure 1 shows the method in accordance with the invention for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane, where the refrigeration necessary for the process takes place by means of a partial stream of the carbon monoxide product fraction.

Figure 2 shows the method in accordance with the invention for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane, where the refrigeration necessary for the process takes place by means of a closed refrigeration cycle.

Figure 3 shows an alternative method to the method of Figure 2 with regard to the closed refrigeration cycle.

In the method shown in Figure 1, the nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane is sent by means of lines 1 and 1' through heat exchangers E1 and E2, respectively. In these heat exchangers the gas stream is cooled by process streams, which will be discussed in more detail below, and a large portion of the carbon monoxide, nitrogen and methane

condenses. Before being sent to the low-temperature process, the gas stream in line 1 is, if necessary, prepurified, for example, in a carbon dioxide wash and, by means of an adsorber station, purified to residual amounts of water that are tolerable in the low-temperature process. The gas stream leaving heat exchanger E2, which is already partially condensed, is sent by line 1" to a separator D. A first hydrogen-rich fraction is withdrawn at the top of separator D, heated by the input gas stream that is to be cooled in heat exchanger E2, sent by means of line 2 to heat exchanger E1, in which a further heating takes place, and then withdrawn from the process by means of line 2'.

According to another advantageous embodiment of the method in accordance with the invention, the first hydrogen-rich fraction that is formed in the partial condensation and is withdrawn by means of lines 2 and 2' can be sent to a post-purification step. This post-purification of the hydrogen-rich fraction can take place, for example, by means of adsorption, especially by means of pressure exchange adsorption, or by means of one or more membrane separation devices, preferably arranged in cascade fashion. The carbon monoxide yield of the method in accordance with the invention is about 85%. Including the post-purification of the hydrogen-rich fraction and a recycling of the membrane retentate connected with that, or a recondensation of the rinse gas of the pressure exchange adsorption that is connected with it, the carbon monoxide yield can be increased to about 97%.

The carbon monoxide-rich condensate containing methane and nitrogen that accumulates in the bottom of separator D is sent by line 3 to an expansion valve a, where it expands and is sent by line 3' to the top of the hydrogen stripping column T1. A second hydrogen-rich fraction is withdrawn at the top of the hydrogen stripping column T1 by means of line 4. This fraction is expanded in expansion valve b and then sent via line 4' to the hot gas path (lines 8, 15, 15' and 15"), which will be discussed in more detail below. The carbon monoxide-rich bottom product of the hydrogen stripping column T1 that contains nitrogen and methane is withdrawn by means of line 5, expanded in expansion valve d and then sent via line 7 to a first

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rectification column T2. To produce the reboiler vapor required in the hydrogen stripping column T1, a partial stream of the bottom fraction withdrawn via line 5 from the hydrogen stripping column T1 is evaporated against the process streams that are to be cooled, in heat exchanger E2.

A nitrogen-rich fraction is withdrawn at the top of the first rectification column T2, expanded in expansion valve c and likewise mixed into the previously mentioned hot gas path. A carbon monoxide-rich bottom fraction containing methane is withdrawn from the first rectification column T2 via line 9. A partial stream of this fraction is sent via line 10 through heat exchanger E3, in which evaporation takes place against the process streams that are to be cooled, in order to generate the reboiler vapor required in rectification column T2. To improve the separation properties in rectification column T2, a nitrogen/carbon monoxide fraction is withdrawn in the upper region via line 30, cooled in heat exchanger E4 and then fed back to the top of rectification column T2 via line 30'.

The already mentioned carbon monoxide-rich, methane-containing fraction withdrawn via line 9 is divided after withdrawal of the part that is necessary to obtain the reboiler vapor. A partial stream is expanded in expansion valve e, sent via line 11 to heat exchanger E2 and evaporated completely in it. Then the now completely evaporated partial stream is sent via line 11' to the middle region of a second rectification column T3 as intermediate heating. The second partial stream is expanded in expansion valve f and then likewise sent via line 12 to the already mentioned second rectification column T3 as intermediate reflux.

A fraction that essentially contains methane accumulates in the bottom of this rectification column T3 and is withdrawn via line 13, expanded in expansion valve g and then sent via line 13' to the hot gas path that was already mentioned. A partial stream of this fraction, which essentially contains methane, is sent via line 14 to heat exchanger E3, in which evaporation takes place to generate the necessary reboiler stream, and then fed back to the lower region of the second rectification column T2.

The streams or fractions that are sent to the hot gas path are additionally purified before the heat exchanger E3. They are then sent via line 15 to heat exchanger E3 in which they are heated, then sent via line 15' to heat exchanger E1 in which they are heated to the desired input temperature, and then withdrawn via line 15" from the process for further use, for example, for undergrate firing of a steam reformer.

A highly pure, carbon monoxide product fraction is withdrawn via line 16 at the top of rectification column T3. It is then, after mixing in other carbon monoxide streams with product purity, which will be discussed in more detail below, sent via lines 17 and 18 to heat exchanger E2, in which a heating takes place by the process streams that are to be cooled. The carbon monoxide product fraction is sent via line 18' to heat exchanger E1, in which additional heating to the desired input temperature takes place. After that, the heated carbon monoxide product fraction is sent via line 18" to a single stage or multistage condenser V and is withdrawn from the process via line 19.

For refrigeration for the method in accordance with the invention, a partial stream of the heated and condensed carbon monoxide product fraction is sent via line 20 to heat exchanger E1, in which said partial stream is cooled by the process streams that are to be heated. Then the cooled carbon monoxide product stream is sent via line 20' to turbine T and expanded in it to produce cold. A bypass line 21 that has a control valve x is provided in parallel to turbine T for amount correction. The carbon monoxide stream expanded in turbine T to produce cold is withdrawn from turbine T via line 22. Now the expanded carbon monoxide stream is divided, where the greater portion is sent via line 23 to heat exchanger E3. Here, it is cooled by the already mentioned process streams in lines 10, 14 and 15 and condensed. Then this carbon monoxide partial stream is sent via line 23 to an additional branching point. A partial stream is expanded in expansion valve k and delivered via line 24 as recycling to the upper part of the rectification column T2.

The second partial stream is sent via line 25 to expansion valve l, in which it is expanded, and then mixed into a carbon monoxide partial stream, which will be discussed in more detail below.

The smaller partial stream remaining after dividing the carbon monoxide stream in line 22 is sent via line 27 to heat exchanger E2. In this heat exchanger it is cooled by the process streams that are to be heated and condensed and then sent via line 27' to a branching point. From this branching point, a partial stream is sent via line 28 and expansion valve h to the already mentioned carbon monoxide product line 17 or 18. This partial stream in line 28 serves to prepare the required peak cold in heat exchanger E2. The other carbon monoxide partial stream is expanded in expansion valve e and then sent via line 29, after prior mixing the carbon monoxide partial stream by means of line 25, and via line 26 to heat exchanger or condenser E4. Then this carbon monoxide stream is sent via line 26' to the already mentioned carbon monoxide product fraction in lines 16 or 17.

Carbon monoxide losses are caused only by the hydrogen-rich, nitrogen-rich or methane-rich fractions that are sent to the hot gas path, each of which has small levels of carbon monoxide. /5

Figures 2 and 3, as already noted, show the method in accordance with the invention for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane, where the refrigeration necessary for the process takes place by means of a closed refrigeration cycle. However, in what follows, only the differences from the method described by means of Figure 1 are discussed.

In the method described in Figures 2 and 3 the carbon monoxide product fraction formed at the top of the rectification column T3 is withdrawn via lines 40 and 42 and sent to heat exchanger E3. Here, a heating takes place by the process streams that are to be cooled, after which the carbon monoxide product fraction is sent via line 42' to heat exchanger E1, in which a further heating to the input temperature takes place, and then it is withdrawn from the process via line 42".

A closed nitrogen refrigeration cycle is used here for refrigeration. It goes without saying that other substances or substance mixtures could be used besides nitrogen as refrigerating agents. In the process described in Figure 2, the arrangement of the nitrogen circulation is by and large identical to the carbon monoxide circulation as shown in Figure 1. After condensation (V) and subsequent cold-producing expansion (T), the refrigerating agent stream is divided into two partial streams (lines 53 and 56). The refrigerating agent stream in line 53 is cooled and condensed in heat exchanger E3 and then combined with the other refrigerating agent stream by means of line 53' and expansion valve m. This stream is sent first via line 56 to heat exchanger E2 and is cooled and condensed in it by the process streams that are to be heated. Then it is sent via line 56' to a branching point, from which a first partial stream is sent via line 57 to an expansion valve h. This partial stream serves to prepare the necessary peak cold in heat exchanger E2. The second partial stream is sent via line 58 to expansion valve i, expanded in it and then, after the already mentioned admixing of the other partial stream from line 53', sent via line 54 to the heat exchanger or condenser E4. In it, it serves for refrigeration for the process streams in lines 41 and 59, which will be discussed below. Then the refrigerating agent stream is sent via lines 54' and 55 to heat exchanger E2 and heated in it. It is then sent via line 55' to heat exchanger E1 in which additional heating takes place, and then it is sent via line 55'' to the inlet of condenser V. The refluxes required for the rectification columns T2 and T3 (line 41, 41' and expansion valve p and lines 59 and 59') is generated in heat exchanger or condenser E4.

Figure 3 shows a variation of the refrigerating agent management described in Figure 2. Here, the division of the refrigerating agent stream no longer takes place right after the cold-producing expansion in expansion turbine T, but rather a partial stream of the refrigerating agent is already sent before turbine T to heat exchanger E2 via line 60. In heat exchanger E2 this refrigerating agent stream is cooled and

condensed and then sent via line 60' to the already described branching point before the two expansion valve h and i.

In general it can be said that refrigeration by means of a partial stream of the carbon monoxide product fraction – as shown in Figure 1, has the disadvantage that the carbon monoxide condenser V is comparably expensive. However, in exchange, the carbon monoxide product fraction can be delivered under pressure. If a closed refrigeration cycle is used, in particular a nitrogen refrigeration cycle, a considerably cheaper condenser V can be used. However, it is disadvantageous with this conduct of the process that the carbon monoxide product fraction in this case accumulates practically under no pressure. To avoid this disadvantage, as shown in Figure 3, the carbon monoxide-rich, methane-containing bottom fraction from rectification column T2 can be pumped to a higher pressure, so that the rectification column T3 can be operated at a higher pressure and the carbon monoxide product fraction withdrawn from the top of rectification column T3 can be delivered at a higher pressure. Since in this case the partial stream of the carbon monoxide-rich, methane-containing bottom fraction, which is expanded via expansion valve e, must be evaporated at a higher pressure, a higher refrigeration circulation pressure is also necessary. This means that the condenser V must achieve a higher output pressure, and the refrigerating agent partial stream that is required in heat exchanger E2 for refrigeration should be not be withdrawn at the outlet of the expansion turbine T as shown in Figure 2, but rather before the expansion turbine T.

The three tables 1-3 below give the compositions of substances and other parameters such as pressure, temperature, etc., of certain selected lines or process streams:

TABLE 1

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	Line 1	Line 2'	Line 3	Line 4	Line 5	Line 8	Line 9
H ₂ [mol%]	47.5	87.4	4.0	70.4	0.0	1.8	0.0
N ₂ [mol%]	0.2	0.0	0.3	0.2	0.3	53.3	0.0
CO [mol%]	51.5	12.5	94.0	29.4	97.9	45.0	98.2
CH ₄ [mol%]	0.8	0.0	1.0	0.0	1.7	0.0	1.8
T [°C]	11.0	36.0	-186.5	-186.7	-170.8	-185.8	-182
p [bar]	20.4	19.9	20.1	6.3	6.4	2.4	2.6
Amount [kmol/h]	325.6	169.7	155.9	8.9	147.1	0.8	146.2
	Line 11'	Line 12	Line 13	Line 15"	Line 16	Line 18	Line 19
H ₂ [mol%]	0.0	0.0	0.0	45.7	0.0	0.0	0.0
N ₂ [mol%]	0.0	0.0	0.0	3.4	0.0	0.0	0.0
CO [mol%]	98.2	98.2	37.0	32.5	99.9	99.9	99.9
CH ₄ [mol%]	1.8	1.8	63.0	18.4	0.0	0.0	0.0
T [°C]	-179.9	-187.2	-179.1	36	-187.6	187.6	40.0
p [bar]	1.7	1.6	1.7	1.6	1.6	1.6	4.3
Amount [kmol/h]	138.2	8.0	4.0	13.7	156.5	428	142.2

	Line 23	Line 27	Line 24	Line 25	Line 28	Line 29	Line 26'
H ₂ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0
N ₂ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO [mol%]	99.9	99.9	99.9	99.9	99.9	99.9	99.9
CH ₄ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0
T [°C]	-159.3	-159.3	-187.6	-182.0	-187.2	187.0	-187.5
p [bar]	3.1	3.1	1.6	3.0	2.9	2.9	1.6
Amount [kmol/h]	222.8	63.0	14.3	208.5	13.6	49.4	257.9
	Line 30'						
H ₂ [mol%]	0.0						
N ₂ [mol%]	44.1						
CO [mol%]	55.9						
CH ₄ [mol%]	0.0						
T [°C]	-185.8						
p [bar]	2.4						
Amount [kmol/h]	261.0						

TABLE 2

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	Line 40	Line 41'	Line 42'	Line 50	Line 53	Line 56
H ₂ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0
N ₂ [mol%]	0.0	0.0	0.0	100.0	100.0	100.0
CO [mol%]	99.9	99.9	99.9	0.0	0.0	0.0
CH ₄ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0
T [°C]	-186.4	-186.4	16.9	40.0	-159.3	-159.3
p [bar]	1.8	1.8	1.4	6.8	4.9	4.9
Amount [kmol/h]	156.5	14.3	142.2	327.9	264.9	63.0
	Line 54'	Line 55'				
H ₂ [mol%]	0.0	0.0				
N ₂ [mol%]	100.0	100.0				
CO [mol%]	0.0	0.0				
CH ₄ [mol%]	0.0	0.0				
T [°C]	-187.6	-165.2				
p [bar]	2.4	2.3				
Amount [kmol/h]	307.1	327.9				

TABLE 3

	Line 9	Line 9'	Line 11'	Line 12	Line 13	Line 40	Line 42''
H ₂ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0
N ₂ [mol%]	0.0	0.0	0.0	0.0	0.0	0.0	0.0
CO [mol%]	98.2	98.2	98.2	98.2	37.0	99.9	99.9
CH ₄ [mol%]	1.7	1.7	1.7	1.7	63.0	0.0	0.0
T [°C]	-182.0	-181.9	-172.5	-181.9	-169.6	-179.7	17.8
p [bar]	2.6	3.6	3.4	3.3	3.4	3.3	2.9
Amount [kmol/h]	146.2	146.2	141.4	4.8	4.0	164.1	142.2

Claims

1. A method for obtaining carbon monoxide from a nitrogen-contaminated gas stream that essentially contains hydrogen, carbon monoxide and methane, in which

- a) the gas stream is cooled and partially condensed,
- b) the first hydrogen-rich fraction that accumulates in the partial condensation is removed,
- c) the carbon monoxide-rich condensate, which contains methane and nitrogen and is obtained in the partial condensation is sent to a hydrogen stripping column,
- d) in said column, separation into a second hydrogen-rich fraction and another carbon monoxide-rich fraction takes place, and
- e) said carbon monoxide-rich fraction containing methane and nitrogen is separated at low temperature into a highly pure carbon monoxide fraction and a fraction essentially containing methane and nitrogen

characterized by the fact that

f) the carbon monoxide-rich fraction that contains methane and nitrogen coming from the hydrogen stripping column is sent to a first rectification column,

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g) in said rectification column, a separation into a nitrogen-rich and a carbon monoxide-rich fraction containing methane takes place,

h) the nitrogen-rich fraction is withdrawn,

i) the carbon monoxide-rich fraction containing methane is sent to a second rectification column,
and

k) in said rectification column, a separation into the highly pure carbon monoxide product fraction and into a fraction essentially containing methane takes place.

2. A method as in Claim 1, characterized by the fact that at least one partial stream of the carbon monoxide product fraction, after condensation and cold-producing expansion, is used for the refrigeration required for the process.

3. A method as in Claim 1, characterized by the fact that the refrigeration necessary for the process takes place by means of a closed (mixture) refrigeration cycle.

4. A method as in Claim 3, characterized by the fact that nitrogen is used as the refrigerating agent for the closed refrigeration cycle.

5. A method as in one of Claims 1-4, characterized by the fact that the first hydrogen-rich fraction obtained in the partial condensation is sent to an post-purification step.

6. A method as in Claim 5, characterized by the fact that the post-purification of the hydrogen-rich fraction takes place by means of adsorption, in particular by means of pressure exchange adsorption, or by means of one or more membrane separation processes that are preferably arranged in cascade fashion.

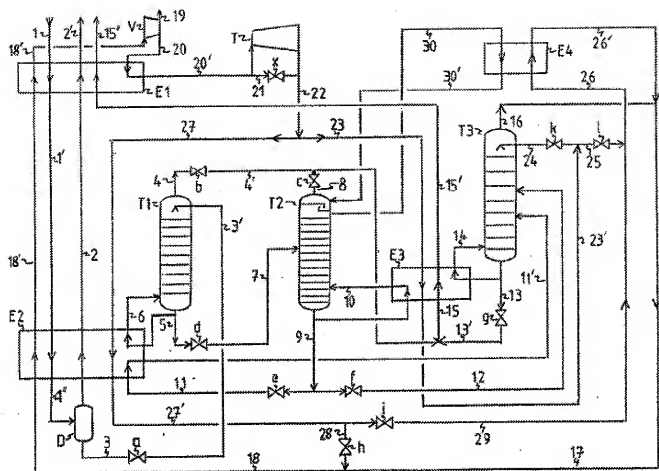


Figure 1

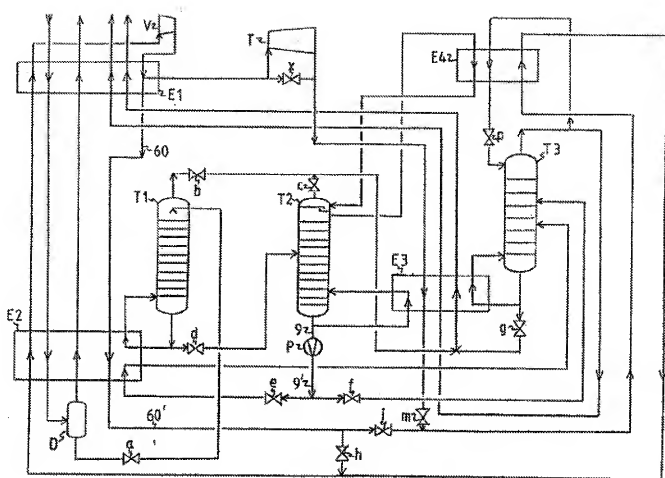


Figure 3